

Observations at Explo Systems Inc., Camp Minden LA.

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On 20 Dec I met with Colonel Ronnie Stuckley, Camp Minden base commander, Lt. John Porter and Investigator Shelley Hopkins, Louisiana State Police (LSP), Jimmy Shaw and Will Grevel, APE 1995/near infrared (NIR) specialists from Joint Munitions Command (JMC), and Michael Kile, quality assurance (QA) and safety manager at Explo Systems. Focus of my observations was sampling and testing procedures for M6 multi-perforated propellant (M6_MP).

Investigator Hopkins has established a testing priority as follows: (1) lots that require moving (2) lots that have been improperly stored (3) lots stored in magazines. Each lot is to be tested. Sampling was being done from the center of the containers, the idea being that grains at that location were likely to experience maximum heating. I pointed out that though this would seem logical, there was no available data supporting that assumption. I suggested that grains be taken instead from random locations within a container, as per conventional practice, so as to assure representative samples. Sgt. Hopkins agreed. Containers (60 lb. boxes, 120 lb. drums and 180 lb. nylon bags) are being sampled twice, each sample consisting of sufficient grains to fill the NIR cell. This is more than adequate. NIR data thus far indicates close agreement between the duplicates. If this trend continues, it might be acceptable to draw only one sample from a given container. I suggested that a limited number of lots be tested on both instruments, to reveal possible instrument bias (consistent low or high results from one instrument compared to the other). A few dual readings were taken,

showing very close agreement. It is recommended this practice be continued. LSP personnel have been trained on the APE 1995 by the JMC staff, have expressed their comfort working with the instruments and appear to be following good laboratory practices. Results as of 20 Jan varied from 0.5 to 0.8 % renaming effective stabilizer (RES), all within Category A. Four lots tested by NIR were analyzed in Expo's chemistry lab; results were in close agreement with NIR data. It was reported that some propellant was visibly wet; these gave erratic NIR readings. After being dried under ambient conditions, they were retested and gave normal results, all Cat. A. Some propellant grains were of unusual color, various shades of blue, green and yellow, sometimes in mottled patterns. Odd colors are not uncommon and are not to be taken as a sign of degradation. JMC personnel were under the impression that the Cat. C threshold for APE 1995 testing was the same as for high performance liquid chromatography (HPLC), the recognized standard technique. This is not the case. Onset of Cat. C is below 0.30%, but NIR results under 0.45% require HPLC confirmation due to lower accuracy of the former method. When significant differences between NIR and HPLC results occur, HPLC overrides. The testing protocol was adjusted accordingly.

Expo's lab HPLC instrument is basically adequate for propellant stabilizer analysis. It is an Agilent Model 1260 Infinity, a widely used system introduced in 2010. The system does however lack an auto-sampler. Mr. Kile does the testing in accordance with a procedure (SOP 61, 07 Jul 2011) written by his former immediate superior, a chemist no longer with Expo. A review of current procedures reveals some serious shortcomings: (1) Contrary to normal laboratory practices, samples are not run in duplicate, hence no estimate of measurement precision is possible. (2) A certified reference analytical standard (from Cerilliant Corporation or some other recognized lab supply house) is run only once, at the start of the 8 hour day shift in which testing occurs; any subsequent instrument drift and associated errors would go undetected. (3) No control or internal standards are run; hence no estimate of instrument accuracy is possible. (4) Only 1 grain per sample is analyzed; since M6_MP is characterized by high grain-to-

grain stabilizer variation within a given lot, results from a single grain may not be representative.

Suggestions for improving chemistry lab procedures are as follows: (1) Run all samples in duplicate, 1 injection each for 2 separate sample preparations; if results are within +/- 5% of each other, average the results, otherwise redo (2) Run 1 analytical standard at the start of the daily run, one at the end; if they are within +/- 5% of each other, average them, if not, repeat entire run (3) Use another analytical standard from a batch other than the 1st one, inject at midpoint of the day's run, analyze it as a sample or "control"; if result is not within +/-5% of the control's certified value, rerun. (4) Use at least 2 grains for each of the 2 recommended sample preparations, yielding a total of 4 grains tested per lot versus 1 under current procedures.

There are some other issues re HPLC results. A review of Explo's HPLC Propellant Stability Report (Form # 158) shows that none of the 6 secondary stabilizer products added into the RES have the necessary correction factors applied to them. These factors adjust for the reduced effectiveness of the secondaries compared to that of the primary stabilizer, diphenylamine (DPA). Failure to apply those causes reported RES values higher than the actual ones. Another potential issue arises from an examination of the HPLC Propellant Stability Report example (Form # 158) shown on P. 10 of the lab SOP. It shows the degradation product 4,4' dinitro-diphenylamine (4,4' DNDPA) at 0.603%, a very high value for this substance, inconsistent with anything previously observed for M6 or any other single base propellant to my knowledge. I don't know the significance of this apparent anomaly, but it should be checked by examining pertinent spreadsheets and chromatograms, so as to better understand Explo's calculations and to insure no gross miscalculation is being made.

Another observation re lab operations that is the HPLC is being run manually; samples are injected into the instrument with a handheld syringe as opposed to using an auto-sampler. Likewise, raw data output (retention time, area count, peak height etc.) is manually entered into an external spreadsheet to calculate

final results, rather than going directly to the system computer. Manual operation precludes overnight runs. Net result is that fewer samples per day can be analyzed than otherwise might be the case. QA may also be impacted, in that some necessary standards and controls may be omitted due to insufficient throughput capacity. Manual data entry is not a problem per se, but may make it more difficult to monitor chem lab QA.

The following recommendations to aid in tracking and documenting both HPLC and NIR testing are offered. Differences between the NIR results for duplicate samples should be recorded and the standard deviation calculated continuously. Standard deviation (s) is a measure of random dispersion or variability. See Chart 1 in accompanying spreadsheet "QA Charts". When the same propellant samples are run on both NIR 1 and 2 as cross checks, both the average and standard deviation of the differences should be calculated continuously. The average value (bias) is an indication of any systematic error, i.e. consistently high or low results from one instrument compared to the other. If both instruments were reading identically, bias would be zero. See Chart 2. NIR and HPLC comparative results also need to be tracked. Depending on its sign, positive or negative, the bias will reveal whether HPLC results are running higher or lower than those of the NIR. Standard deviation should be no more than 0.075%. If higher, look for results with high differences and retest. See Chart 3. Results shown in all three charts are examples for illustrative purposes only. All this data compilation may seem like a great deal of work, but in fact should require only a few extra columns, with appropriate embedded math functions, on whatever NIR spreadsheet or sheets are currently in use. NIR operators would not have to enter any additional data or perform any calculations. The benefit would be an audit trail documenting systematic QA monitoring. This is the only way a reasonable level of confidence in test results can be achieved and demonstrated.

Copies of Explo's HPLC spreadsheets and chromatograms have been requested to enable better understanding of how the company calculates stabilizer level. Absent such understanding, it will be impossible to correlate NIR and HPLC data in

a meaningful way. It may be necessary to get additional information from Mr. Kile, depending on what we learn from the documentation received.

It would be useful to determine if the recent explosion of flake and ball propellant, along with M6, was initiated by the former or the latter. Sgt. Hopkins said this has not yet been determined. He agreed to provide ARDEC a copy of the video capturing the explosion, to see if it will provide us with some insight.

Some of the M6 propellant sold to Explo by JMC during the last 2 years has been identified; lot numbers and amounts have been sent to ARDEC for review of stability history. Purpose of the review is to screen for any lots that might be “at risk”. The amount of propellant covered by these lots is only a small portion of the total at Explo; more lot numbers are expected to be identified and forwarded. POC is Ms. Debbie Carstens, JMC, DSN 793-4636.